Feasibility Studies on Usage of Agro-Forestry-Based Ash as a Cement Replacement in Concrete

by

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Abstract

This study is part of a joint Canada-India research program to effect sustainable construction. Alberta generates a significant amount of waste from agriculturaland forest-based industries. Similarly, India has lots of sugar cane bagasse waste. Researchers in both countries want to use these forms of waste in concrete, not only to solve the problem of land filling, but also to use it, if possible, as a supplementary cementing material to reduce the economic and environmental cost of concrete. In the present investigation, a feasibility study is made to examine agro-based ashes as supplementary admixtures in concrete. These agro-based ashes are waste generated from pulp and paper mills, typically consisting of a mixture of hardwood and softwood barks and their fine residues. Also, ash resulting from sugar cane bagasse, which is burnt as fuel in sugar mills, has also been investigated as a pozzolanic admixture, especially for resistance to sustained elevated temperatures. All of the ash samples were first characterized for chemical composition and physical properties, including grain-size distribution, density and chemical composition. Subsequently, the ash was added to concrete as a supplementary cementing material to study the compressive and tensile performance of hardened concrete. With sugar cane bagasse ash, specimens of concrete incorporating different percentages of ash as a mass replacement of Portland cement were subjected to compression and split tensile tests under different temperatures. From the strength point of view, the results are encouraging: for example, when the dosage level of Alberta's agro-based ashes

was as high as 15%, the strength was still seen as promising. The findings strongly endorse that sugar cane bagasse ash imparts resistance to concrete against elevated temperatures and may be used as a supplementary cementing admixture. Results show that these agro-based ashes can be used in normalstrength concrete buildings.

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Chapter 1. Introduction

1.1 Background

These days researchers are studying different agro-based waste materials. The major quantities of waste generated from agricultural sources include sugarcane bagasse, rice husk, and coconut husk. Reusing such waste as a sustainable construction material seems to be a suitable solution for the problem of landfilling and the high cost of building materials (Rabi et al 2009). These days, agricultural solid waste materials are being reused in manufacturing blended mortars and concrete for better performance and price. The use of waste materials in concrete has been a continuous procedure in different industries. These waste agricultural materials are used to heighten the mechanical properties of a building material while they are not useful in agriculture fields. (Cooper et al. 1999, Madurwar et al. 2013, Singh. 2013). Rice husk ash (RHA), sugarcane bagasse ash (SCBA), sawdust and cork granules have been used as pozzolanic materials for reactivity in concrete. They are added to concrete to partially replace cement in concrete mixtures so that the concrete will have sufficient engineering physical and chemical properties (Paya et al. 2002). RHA has been used as a suitable raw material to make hydraulic cement and as a good corrective admixture to reduce expansion that occurs as a result of an alkali-silicate reaction, and also to reduce the temperature in high-strength concrete (AI-Khalafand et al 1984). SCBA is a valuable cement-replacing material for cement and concrete production because of its high content of silicon and aluminium oxides. Also, the effect of elevated

temperature on strength of normal and high strength concrete with and without different ashes is investigated by different researchers.

1.1.1 Use of agro-sourced ash in concrete

In tests on SCBA concrete, Chusilp et al. (2009) found that concrete specimens containing 10-30% ground bagasse ash instead of cement in binder had greater compressive strength than the reference concrete (concrete without ground bagasse ash) at 28 days, while the water permeability of this concrete was lower than that of reference concrete. Comparing different percentages, concrete containing 20% ground bagasse ash had the highest compressive strength at 113% of the reference concrete. Radke et al. (2012) found 20% optimum value and discovered that SCBA can potentially be sold at a price similar to that of slag and fly ash, thereby making it cost effective and environmentally friendly. Puala et al. (2010) got the same 20% optimum value of SCBA in Brazil while there are a lot of sugar cane sources. Findings by Chusilp et al. indicate that ground bagasse ash can be used as a pozzolanic material in concrete to have an acceptable strength, lower heat evolution, and reduced water permeability with respect to the control concrete. Srinivasan et al. (2010) have done similar studies on SCBA concrete and concluded that bagasse ash mainly contains aluminum ion and silica. It has been chemically and physically characterized, and partially replaced in the ratio of 0%, 5%, 10%, 15%, 20% and 25% by the weight of cement in concrete. Fresh concrete tests including a compaction factor test and slump test were done, as were hardened concrete tests including compression, splitting tensile and flexural tests at seven and 28 days. The results showed that the strength of concrete

increased as the percentage of bagasse ash replacement increased. Amin (2011) concluded that the specific surface area of bagasse ash is found to be three times higher than that of cement. The density, specific gravity and mean grain size of bagasse ash are found to be less than those of cement. Kawade et al. (2013) found that SCBA made concrete stronger and easier to work with. Muangtong et al. (2013) found that at older ages, the result is observed that the intensity of CH phase decreases whereas that of C-S-H phase increases in the cement replaced with SCBA comparing to without SCBA. Ultra-finely ground SCBA was produced by vibratory grinding resulted in the production of high-performance concrete with 20% replacement instead of Portland cement Fairbairn et al. (2012). The addition of SCBA also resulted in improvements in the rheology of concrete in the fresh state and resistance to the penetration of chloride-ions. Also, Rukzon et al. (2012) noticed an improvement in concrete's resistance to chloride penetration when SCBA was used to enhance the precipitation sites of hydration products and reducing the Ca(OH)₂ of concrete. Fairbairn et al. studied the thermal, chemical and mechanical behavior of concrete containing 5 to 20% of ash and discovered the viability of possible CO₂ emissions reductions for cement manufacturing. Castaldelli et al. (2013) assessed SCBA blends for the production of alkali-activated pastes and mortars. They found that SCBA is an interesting source for preparing alkali-activated binders.

Ganesan et al. (2008) have studied the effect of rice husk on the strength and permeability properties of concrete. In their study, they looked at RHA prepared from the boiler burnt husk residue of a particular rice mill as a blending component in cements. They analysed the physical and chemical characteristics of RHA and found that it consists of 87% of silica. Test results showed that up to 30% of RHA could be used instead of cement without adversely affecting the strength and permeability properties of concrete. Ramasamy (2010) investigated the effect of rice husk in concrete and concluded that the strength of the concrete increased with increasing replacement percentage of 10% of RHA. Adding 20% RHA increased resistance against sulphate attack for both continuous soaking and cyclic conditions. Adding RHA significantly improves concrete's strength and durability properties. Ramezanianpour et al. (2009) studied the effect of RHA and found that concrete incorporating RHA had higher compressive strength, splitting tensile strength and modulus of elasticity at various ages than the reference concrete.

1.1.2 Effect of elevated temperature on concrete

Rao et al. studied RHA in concrete under elevated temperatures and found that using RHA in concrete is not only cost effective but also improves resistance against elevated temperatures and durability and reduces carbon dioxide emissions. Harison et al. (2014) investigated the effect of fly ash in concrete on strength of Portland cement concrete. In this study, cement was replaced by fly ash in the range of 0% (without fly ash), 10%, 20%, 30%, 40%, 50% and 60% by mass fraction of the cement. It was observed that using 20% fly ash as a replacement increased the strength marginally (1.9% to 3.2%) at 28 and 56 days respectively. Also, when up to 30% of fly ash is used to replace Portland cement, after 56 days the strength is almost equal to that of the reference concrete.

Elsageer et al. (2009) studied the effect of fly ash (FA) and found that FA concrete could be used when early age strength is required. The FA early age strength was found to be equivalent to that of Portland cement concrete. While the age and curing temperature is increased, the strength increases more and is greater than that of the target concrete. However, the long-term strength is adversely affected by higher curing temperatures and by increasing the fly ash. Shang et al. (2013) studied FA in high performance concrete under elevated temperatures and found that while the temperature increased from 200-500°C, the flexural and compression strength dropped sharply in comparison with the original strength. There are also some studies on the effect of elevated temperature on normal and high strength concrete. Arioz (2007) studied the effects of elevated temperatures on properties of concrete and noticed that the surface cracks became visible when the temperature reached 600°C. The cracks were very clear at 800°C and increased extremely when the temperature increased to 1000°C. Concrete specimens subjected to temperatures of 1200°C were completely decomposed. Rao et al. (2004) studied the variation of compressive strength of high-strength concrete at elevated temperatures and observed a gradual increase in compressive strength in the range of 100°C to 400°C and a gradual decrease in compressive strength in the range of 400°C to 700°C. Pathan et al. (2012) concluded that when the temperature is increased to 300°C the compressive, splitting tensile, flexural strength and impact strength of specimens is decreased for conventional and highstrength concrete. Husem (2006) studied the effects of high temperature on the compressive and flexural strength of ordinary and high-performance concrete and concluded that in ordinary concrete and high-performance concrete exposed to high temperatures, flexural and compressive strength decreases when the temperature increases .

1.2 Outline of thesis

In the present research, a feasibility study was made to investigate the potential use of forest-based ashes as supplementary admixtures in concrete. The forestbased ashes are waste from wood suppliers, typically a mixture of hardwood and softwood bark and fines. Chemical and physical tests of XRD, SEM, EDX, XRF, particle and sieve analysis were conducted to characterize the ash for their different chemical and physical properties and potential pozzolanic activity properties.

Subsequently, the potential to use ash as a pozzolanic admixture was examined by replacing Portland cement with the ash from 0 to 20% by mass at 5% increments. This research is designed to study the short-term mechanical performance and fresh properties of concrete. The concrete produced was tested for compression and splitting tension.

In the present investigation, a feasibility study was made to utilize SCBA as an admixture in concrete and examine its role in imparting resistance under elevated temperatures. The ash was obtained from a sugar mill in India where the bagasse was recycled as fuel for the mill. Bagasse is the fibrous matter that remains after sugarcane or sorghum stalks are crushed to extract their juice. It is currently used as a biofuel and in the manufacture of pulp and building materials. For each 10 tons of sugarcane crushed, a sugar factory produces nearly 3 tons of wet

bagasse. Since bagasse is a by-product of the sugar cane industry, the quantity of production in each country is in line with the quantity of sugarcane produced. The sugarcane can be burnt and ground to requirements and used as a cement replacement in concrete.

The background review showed that research work has been carried out to show how SCBA affects the strength of concrete. However, no attempt has been made to explore SCBA as a refractory material. Other agro-based ashes are used in concrete and the effect of temperature on them has been studied. Rice husk ash and fly ash are used in different percentages in concrete, and studies have looked at their performance under compression with exposure to elevated temperatures. This project is designed to examine ash's suitability and effectiveness under elevated temperatures when ash is used as a supplementary cementing admixture. The purposes of this research are: minimizing the usage of cement, thereby reducing the effect of CO₂ on the environment; useful consumption of an industrial waste and facilitating waste management; and studying the variation of compressive strength of cement mortar and cement concrete cubes and flexural strength of cement concrete beams subjected to sustained elevated temperatures. In this study, ash was characterized for its physical properties and chemical composition. Incorporated as a supplementary cementing admixture, SCBA replaced Portland cement from 0 to 25% by mass fraction at 5% increments. The resulting concrete was subjected to elevated temperatures of 300°C, 400°C and 500°C, exposed for 2 hours in each case. A reference series was examined at room temperature. Compressive and flexural strength were evaluated and compared

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with the reference performance at room temperature and reported as residual properties.

Chapter 2. Physical and Chemical Properties of FBAs as Pozzolanic Admixtures in Concrete¹

2.1 Abstract

This thesis is based on a feasibility study that used forest-based ashes as supplementary admixtures in concrete. The forest-based ashes (FBA) are waste generated from burning wood residues, typically consisting of a mixture of hardwood and softwood barks and their fine residues, which are burnt to produce the ash. In the study reported here, the ash was characterized by chemical composition and physical properties. The chemical and physical characterization of the four different types of ash was done by examining each type of ash for grain-size distribution, density and chemical composition. The results of particlesize analysis show that all the forest based ashes had a mean particle size between 100 and 1000 microns, which is larger than that of the Type GU Portland cement used in this study. The chemical characterization from X-ray fluorescence showed that the FBA specimens were predominantly composed of CaO, with significant amounts of SO₃. The chemical analysis further revealed that all specimens contain K₂O and Na₂O at levels which exceed maximum limits on alkali oxides allowed by ASTM and pose concerns about potential alkali-silica reactions. The SO₃ presence also indicates the potential danger of sulphate attack damaging the concrete over the long term, thus affecting the durability.

¹ Parisa Setayesh Gar, Vivek Bindiganavile

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2.2 Introduction

The current research is based on the study of the potential usage of forest-based ash instead of cement as a supplementary material in concrete mixes. As part of the research, different chemical and physical tests are conducted on the ash.

The major quantities of waste from agricultural sources generated in boilers come from sugarcane bagasse, rice husk and coconut husk. Reusing such waste as a sustainable construction material is a good solution for the problem of land-filling and high cost of building materials (Rabi et al 2009). These days, agricultural residue ash materials are often used to manufacture blended mortars and concrete for better performance and price. The use of waste materials in concrete has been a continuous procedure in different fields. These waste agricultural materials are used to heighten mechanical properties of a building material while they are not useful in agriculture fields (Rabi et al. 2009, Cooper et al. 1999, Madurwar et al. 2013, Singh 2013).

Rice husk ash (RHA), sugarcane bagasse ash (SCBA), sawdust and cork granules have been used as pozzolanic materials for reactivity in concrete. They are added to the concrete mixture as partial replacements for cement to ensure sufficient engineering physical and chemical properties (Paya' et al. 2002). RHA has been used as a suitable raw material for making hydraulic cement, as a good corrective admixture for reducing expansion due to alkali-silicate reactions, and to reduce the temperature in high-strength concrete (AI-Khalaf et al. 1984). Also, SCBA is a valuable cement-replacing material for cement and concrete production due to its high content of silicon and aluminum oxides (Paya'et al. 2002). In the present study, XRD (X-ray Diffraction), SEM (Scanning Electron Microscopy), EDX (Energy-dispersive X-ray spectroscopy), XRF (X-Ray Fluorescence), particle and sieve analysis tests were conducted on wood-ash mixtures to determine chemical and physical properties and potential pozzolanic activity.

2.3 Materials and Methods

Four types of ashes with different combinations and densities of 404, 522, 471 and 320 kg/m³ were analyzed with different tests of XRD, SEM, EDX, XRF, sieve analysis and particle size analysis which were produced by other companies. The forest-based ashes are hog fuels from wood suppliers. They are a mixture of hardwood and softwood bark and fines. The hog fuel is from a variety of sources: sawmill bark (mostly softwood), hardwood bark and scraps from the bush from the chipper operations, old hay from surrounding farms, and fibre from the effluent clarifer (primary and secondary sludge). The forest-based ashes were obtained after burning at around 400-470°C. The source of the ashes is not entirely clear but they are from various Alberta based sources. To gather more information about the ashes, particle size analysis and oxide compositions were conducted.

2.4 **Results and Discussions**

As was noted, four types of forest-based ashes were studied. Chemical characterization involving identification and quantification of the chemicals, especially oxides present in the FBA, were done through Energy-dispersive X-ray spectroscopy (EDX), x-ray diffraction (XRD) and x-ray fluorescence (XRF). The

crystallinity and phase analysis of the forest-based ashes were investigated using XRD. XRD test is done to know about the crystal particles of the sample. EDX test is done through elemental analysis of sample and XRF test shows the oxide composition content of the sample and both tests quantify the oxides identified in the ash samples.

Figure 2-1, Figure 2-2, Figure 2-3 and Figure 2-4 show XRD traces of the four different types of FBA used in this study. Table 2-1 gives the various oxides found in the FBA. Further, it was seen that in all cases of ashes (FBA 1 through 4), the chemicals portlandite, arcanite, quartz and calcite exhibited distinct peaks as compared to the other minerals listed in the table below.

Table 2-1- Chemical composition of forest-based ashes as found using XRD

Anhydrite	Quartz	Calcite	Arcanite	Sylvine	Moissanite	Hematite
Apatite	Siderite	Muscovite	Portlandite	Paragonite	Laumontite	Magnesite

XRD outputs shows that quartz and calcite have the most crystallinity. Some of the minerals identified, such as arcanite, are the same as for the Portland cement clinker. Others, such as calcite, quartz and hematite, are the same as for blended Portland cement. Still others, such as portlandite, resemble those found in hydration products (Aranda et al., 2012). Nair et al. (2008) showed that to produce a highly reactive property ash has to contain the maximum amount of amorphous quartz (silica), however, only crystalline phases were detected here.



Figure 2-2- XRD Test of FBA 2







Figure 2-4- XRD Test of FBA 4

Results regarding SCBA by Paya et al. 2002 found that SiO_2 is the mineral in the ash which has a high crystallinity. The highest peaks are for SiO_2 (quartz) and mullite ($2SiO_2.3Al_2O_3$) in the ash, as shown in Figure 2-5. For this thesis, SCBA was tested for its chemical composition and results showed higher levels of SiO2 than of other components.



Figure 2-5- XRD Test of SCBA (Paya', J.et al. 2002)

Previous studies on rice husk ash by Folletto et al. (2006) and Larbi et al. (2010), found that the crystalline structure or the amorphous structure of silica directly depends on the method of burning and the temperature of burning to get the ash. Ramezanianpour et al. (2009) have reported that when the temperature was higher than 650 °C, the rice husk ash contained more crystalline silica. The reactivity decreases when the temperature is increased from 650 and higher. Figure 2-6 below, developed by Larbi et al. (2010), shows the 2-theta range of 18 to 30 degrees in which the silica in rice husk ash may be identified as in an amorphous state.



Figure 2-6- XRD Pattern of As-Received RHA (Larbi et al 2010)

Table 2-2, Table 2-3, Table 2-4 and Table 2-5 below show the percentages of various chemicals that were found using EDX tests in the four different FBAs, FBA1, FBA 2, FBA 3 and FBA 4 respectively. In each case, the Ca and C percent is dominant, suggesting the presence of Ca $(OH)_2$ and Ca (CO_3) . Further, the next dominant elements are K and silicon in FBA 1, 2, 3 and 4. In other words, in these ashes the minerals portlandite, arcanite, calcite and quartz appear to be more predominant than the other minerals listed. The oxide composition shows that the results from EDX and XRF tests in Table 2-8 match.

Table 2-2- EDX Test of FBA 1

Elements	Weight
	Percentage
0	21.58
K	4.78
S	1.20
Al	0.14
Р	0.26
Cl	0.33
Ca	7.11
Si	0.36
Mg	0.66
Na	0.87
С	62.65

Table 2-3- EDX Test of FBA 2

Elements	Weight
	Percentage
0	21.06
K	3.92
S	0.96
Al	0.05
Р	0.12
Ca	4.7
Si	0.25
Mg	0.34
Na	0.15

Table 2-4- EDX Test of FBA 3

Elements	Weight
	Percentage
0	52.97
Ca	26.44
K	7.02
S	2.58
Si	4.59
Mg	1.95
Al	1.35
Na	0.86
Fe	1.08
Р	0.64
Cl	0.24

Elements	Weight
	Percentage
0	36.63
Са	10.11
S	1.46
Si	0.45
Mg	0.96
Al	0.18
Na	0.51
Fe	0.08
Р	0.25
С	47
Cu	0.12
Zn	0.12
K	1.62

However, in FBA 4, CaO is predominant. Results of previous researchers as reproduced in Table 2-6 and Table 2-7 show that the main constituent in rice husk ash is silica, typically 85-88%; the ash also contains some minor oxides such as alkalis, sulphate and portlandite. Similarly, in SCBA, Paya et al. found that silica is the most predominant oxide (2002). Results also showed that the silica remained in the amorphous form between 450-700°C for 3 to 4 hours of husk burning. Since the crystalline forms of silica have less reactivity than the

amorphous one, it is essential that rice husks are burned at a temperature of less than 850°C. The burning temperature of forest-based ashes was around 400-470°C.

Temperature	Loss				Percentage	oxide	composition					
and	on											
Time of Burning	Ignition	SiO2	K20	SO3	CaO	Na2O	MgO	AI203	P205	CI	Fe203	MnO
450°C for 2 hrs	3.49	85.88	4.1	1.24	1.12	1.15	0.46	0.47	0.34	0.39	0.18	0.091
500°C for 2 hrs	3.3	86.89	3.84	1.54	1.4	1.15	0.37	0.4	0.35	0.45	0.19	0.087
550°C for 2 hrs	2.89	87.19	4.1	1.54	1.3	1.05	0.43	0.37	0.32	0.33	0.17	0.091
600°C for 2 hrs	2.69	86.02	3.76	1.82	1.12	1.15	0.39	0.36	0.3	0.27	0.16	0.086
700°C for 2 hrs	2.38	85.81	4.1	1.88	1.4	1.22	0.4	0.38	0.3	0.14	0.17	0.091
850°C for 2 hrs	1.89	87.72	3.96	1.25	1.43	1.11	0.36	0.4	0.3	0.16	0.16	0.09

Table 2-6- Chemical Analysis of Rice Husk Ash (AI-Khalaf et al., 1984)

Table 2-7- Chemical Composition of SCBA (Paya et al. 2002)

LOI	SiO2	AI203	Fe203	CaO	MgO	SO3	Na2O	К2О
0.63	59.67	20.69	5.76	3.36	1.87	1.06	1.11	1.37

Oxide:	FBA 1	FBA 2	FBA 3	FBA 4	
	Average Wt%	Average Wt%	Average Wt%	Average Wt%	
Na2O	2.45	1.92	2.99		
MgO	5.81	3.45	5.21	1.94	
AI2O3	1.98	2.50	1.78	0.53	
SiO2	11.88	14.31	10.31	2.31	
P2O5	4.09	3.16	3.60	3.43	
SO3	25.60	28.03	31.02	18.47	
К2О	9.94	13.93	13.24	8.10	
CaO	36.00	29.99	29.72	63.03	
TiO2	0.22	0.28	0.19	0.22	
MnO	0.18	0.19	0.16	0.24	
Fe2O3	1.37	1.85	1.25	1.01	
Ni2O3	0.02	0.01	0.01		
CuO	0.02	0.01	0.01	0.02	
ZnO	0.30	0.23	0.39	0.50	
Br2O	0.01	0.01	0.01	0.02	
SrO	0.12	0.12	0.11	0.13	

Table 2-8- Chemical Composition of Forest Based Ashes

Regarding the different FBA mineral compositions, it can be seen that FBA 4 has a higher CaO content than the other ashes, which may lead to a latent hydraulic effect. A scanning-electron microscope (SEM) was used to test the forest-based ash specimens for their particle shape and size.

Also, SEM was used to study the ashes to observe their particles, particularly their size. Nine particles of FBA 1, 6 particles of FBA 2 and 3 particles of FBA 3 were tested. The particles in FBA 1, FBA 2, FBA 3 and FBA 4 were more than 100 microns and shapes were as follows: FBA 1 particles were spherical and book-structured, FBA 2 particles were pin-like and spongy, FBA 3 particles were spherical, non-spherical and sheet-like and FBA 4 particles were spherical,

spongy and book-structured. The shapes of the ash particles are shown in Figure 2-7, Figure 2-8, Figure 2-9 and Figure 2-10.



Figure 2-7- SEM Test of FBA 1



Figure 2-8- SEM Test of FBA 2



Figure 2-9- SEM Test of FBA 3



Figure 2-10- SEM Test of FBA 4

Particle-size analysis of the forest-based ashes was done by sieve analysis and particle-size analysis by Mastersizer instrumentation as shown in Figure 2-11. Laser diffraction measures particle-size distributions by measuring the angular variation in the intensity of light scattered when a laser beam passes through a dispersed particulate sample. The angular scattering intensity data is then analyzed to calculate the size of the particles responsible for creating the scattering pattern, using the Mie theory of light scattering.



Figure 2-11- Mastersizer and Sieve Vibrator

Figure 2-12, Figure 2-13, Figure 2-14 and Figure 2-15 show the distribution of the particles for FBAs 1 through 4 respectively. The FBAs are compared with one another in Figure 2-16.



Figure 2-12-FBA 1 Particle Size



Figure 2-13-FBA 2 Particle Size



Figure 2-14- FBA 3 Particle Size



Figure 2-15- FBA 4 Particle Size



Figure 2-16- Comparison of Particle-Size Analysis

The comparative plots in the figure above show that FBA 4 has more of the finer particles while FBA 2 has the coarsest particle-size distribution. Since finer particles ensure a better strength performance both by acting as filler and as a pozzolanic material in concrete, FBA 4 was at this stage in the study thought to be

the best suited of the four FBA samples for use as a pozzolanic replacement for cement. Based on paper by Tran H.N. et al (1987), while Na/ (Na+K) is increasing, the temperature of the boilers is decreasing and this is clear in FBA ashes examined here. Also, while this ratio is lower for some of the ashes, the particle size is lower too, which results in lower probability of alkali-silica reaction based on Zerbino et al. (2012) as well ashigher strength in concrete.

Paya et al. (2002) tested SCBA particles using SEM and found that they were between 34.2 and 36.8 um, making them smaller than FBA. Ganesan et al. (2007) determined that particles can be between 1 and 100 um. These sizes are shown in Figure 2-17. Ganesan found fibrous particles, carbonaceous particles, prismatic particles and air-bubbles particles. RHA particles are made up of a large number of button-like structures, bumps and micropores between 1 and 8 μ m, as shown in Figure 2-18 (Yuzer, 2013). RHA was prepared by burning the rice husk for two hours at 600 °C (Shatat, 2014).



Figure 2-17- Particle-Size Distribution Curves of OPC and BA [Ganesan, A. et al. 2007]


Figure 2-18- Grading Curve of RH. (Yuzer 2013)

Based on ASTM requirements, results show that the sum of silica, alumina and iron oxides in FBA 1, 2, 3 and FBA 4 is lower than the minimum allowable percent and ashes' value of Alkalis is more than its allowable maximum limit. In other words, the ashes don't obey the minimum requirements of ASTM C618, but regarding the findings of Zerbino et al. (2012), if the ash particles become smaller than 75 microns, the potential of alkali silica reaction would be less.

2.5 Conclusions

The forest-based ashes were chemically and physically investigated and studied through XRD, XRF, EDX, SEM and particle-size analysis. Based on the observations made in this study, the specific findings were made:

- 1. It was noticed that in the ashes, FBA 1 through 4, the chemicals portlandite, arcanite, quartz and calcite exhibited distinct peaks compared to the other minerals.
- 2. FBA 4 has more of the finer particles while FBA 2 has the coarsest particle-size distribution. FBA 4 was considered the best-suited of the four

FBA samples because of their fine particles for use due to their filler effect and as a pozzolanic replacement instead of cement.

- 3. The size of the particles in FBA 1, FBA 2, FBA 3 and FBA 4 were more than 100 microns. The shapes of the particles in these ashes are as follows: FBA 1 particles were spherical and book-structured; FBA 2 particles were pin-like and spongy; FBA 3 particles were spherical and non-spherical and sheet-like; and FBA 4 particles were spherical, spongy and book-structured.
- 4. With respect to different FBA mineral compositions, it is inferred that in FBA 4, the CaO content is more than the other ashes which may potentially impart a latent hydraulic effect.

2.6 References

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Chapter 3. Investigation of hardened properties of concrete with supplementary forest-based ashes²

3.1 Abstract

This paper reports the results of an investigation conducted on concrete containing a partial replacement of type GU Portland cement with forest-based ash (FBA) as a pozzolanic admixture. The present investigation, a feasibility study, is made to utilize forest-based ashes as supplementary admixtures in concrete. The forest-based ashes are waste from wood suppliers, typically consisting of a mixture of hardwood and softwood barks and their fine residues, which are burnt to produce the ash. In the study reported here, the ash was first characterized for chemical composition and physical properties. The chemical and physical characterization of the four different types of ash was done by examining the ashes for grain-size distribution, density and chemical composition as described in the previous chapter. Subsequently, the ash was added to concrete as a supplementary material to study the tensile and compressive strength properties of hardened concrete. Specimens of concrete incorporating from 5 to 20% of ash as a mass replacement of Portland cement with FBA were subjected to compression and split tensile tests. Although FBAs contain SO3 and alkalis, which will cause alkali reaction as discussed in the previous chapter, from the strength point of view, the results are encouraging in as much as at low dosage of the FBA, the compressive strength in fact showed an increase over that of the reference mix with zero FBA. At a 15% dosage level of FBA, the strength was

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still seen to be the same as that of the reference mix. With regard to the tensile strength, it is also seen that there is no significant change compared to the reference mix, regardless of the ash content.

3.2 Introduction

The work reported here is from a study conducted to ascertain the performance of concrete containing FBA as a partial pozzolanic replacement of Portland cement. The FBA was obtained by burning forest wood barks, both hard and soft wood, and fines that are deemed waste and so used as fuel in the pulp and paper industry in the western Canadian province of Alberta.

This study focused firstly on characterizing the ash samples for physical and chemical properties, and secondly on the short-term mechanical performance of concrete made with the ash as a supplementary cementing admixture.

The possibility of gainfully using agricultural waste in concrete mixes both to solve the waste disposal problem and to obtain a cost advantage in concrete production has caught the attention of researchers in the last couple of decades. The major quantities of waste generated from agricultural sources are sugarcane bagasse, rice husk and coconut husk. Using such wastes as a sustainable construction material is a suitable solution for pollution and the land-filling problem and high cost of building materials. Accordingly, sugarcane bagasse ash (SCBA), rice husk ash and coconut husk ash have variously been examined for their pozzolanic potential. (Rabi et al 2009) These days, the use of agricultural solid waste materials in concrete and mortars has been a continuous procedure in different fields. These waste agricultural materials are used to heighten

mechanical properties of a building material while they are unused in agriculture fields. A brief survey of available research shows that SCBA and rice husk ash have both been examined at length.

Puala et al. (2010) focused on evaluating the effects of the partial replacement of Portland cement by sugar cane bagasse ash (CBC) in mortars. The main objective was to find a suitable destination for the increasing amounts of residual waste being generated in Brazilian sugar mills. The results from tests with mortars indicated the viability of the partial substitution of cement by up to 20% of the CBC considered.

Fairbairn et al. (2012) studied the thermal, chemical and mechanical behavior of concretes containing 5 to 20% of SCBA and indicated that SCBA improved the performance of all the properties analyzed, and that ash can be used as a supplementary cementing admixture. In a recent study, Castaldelli et al. (2013) assessed SCBA blends for the production of alkali-activated pastes and mortars. It was demonstrated that SCBA is a source for preparing alkali-activated geopolymers.

Chusilp et al. (2009) investigated SCBA concrete and found out that ground bagasse ash can be used as a pozzolanic material in concrete with an acceptable strength, lower heat evolution, and reduced water permeability with respect to the control concrete. Concrete containing 20% ground bagasse ash had the highest compressive strength at 113% of the control concrete. Srinivasan et al. (2010) conducted similar studies on SCBA concrete and concluded that bagasse ash mainly contains aluminum ion and silica. Bagasse ash was replaced instead of cement in the ratio of 0%, 5%, 10%, 15%, 20% and 25% in mass fraction. The result shows that the strength of concrete increased as the percentage of bagasse ash replacement increased. Radke et al. (2012) studied the effects of SCBA and concluded that bagasse ash can increase the overall strength of concrete when used up to 20% as supplementary material instead of cement. They have reported that this ash is a valuable pozzolanic material and it can potentially be sold at a price similar to that of slag and fly ash, thereby making it cost effective and environmentally friendly.

Ganesan et al. (2008) studied the effect of rice husk ash (RHA) with different replacements for strength and permeability properties of concrete under different temperatures. Also Ramasamy (2010) investigated the effect of rice husk ash in concrete and concluded that replacement of 10% increased strength 7.07% at 90 days compared to normal concrete. Also, Ramezanianpour et al. (2009) studied the effect of RHA. Results showed that concrete incorporating RHA had greater compressive strength, splitting tensile strength and modulus of elasticity at various ages than the reference unblended concrete.

Although a number of researchers have studied the ashes from agricultural waste, there appears to be no information available on the potential for ash sourced from using forest waste such as wood bark. A considerable amount of such ash is produced when the low quality wood products deemed unfit for use in the pulp and paper industry are instead used as fuel. This study was undertaken principally to assess the effect of using such ashes as a pozzolanic admixture in concrete. They seem to be cost effective, so they are used in concrete instead of cement and they bring compressive and tensile strength around normal concrete strength, so they can be used in normal strength concrete buildings. This study looks at using FBA to partially replace cement in concrete specimens with percentages varying from 0 to 20 (i.e., 0%, 5%, 10%, 15%, 20%) under normal temperature. It also looks at concrete's compressive and tensile strength performance.

3.3 Experimental Program

Concrete cylinders were prepared with varying amounts of forest-based ash. Four different types of ash derived from burning forest waste in Alberta's pulp and paper mills were used. For each type, specimens were made, with the dosage varying 5 to 20% in steps of 5%. A reference mix was also prepared containing only cement, sand, aggregates and water. Thus, in all, twenty mixes were examined and the samples were subjected to compression and split tensile tests using the appropriate test equipment.

3.3.1 Materials

Type GU Portland cement conforming to ASTM C150 and C595 and locally available dry coarse aggregate and sand conforming to ASTM C33 were used to cast the specimens. Materials used in the test are as follows: GU cement with a bulk density of 1450 (Kg/m³); coarse aggregate with 8mm size and bulk density of 1710 (Kg/m³); and fine aggregate with a bulk density of 1900 (Kg/m³). The four types of ash, FBA1 through FBA4, had bulk densities, respectively, of 404

(Kg/m³), 522 (Kg/m³), 471 (Kg/m³) and 320 (Kg/m³). The forest-based ashes are hog fuel from wood suppliers. They are a mixture of hardwood and softwood bark and fines. The hog fuel is from a variety of sources: sawmill bark (mostly softwood), hardwood bark and scraps from the bush from the chipper operations, old hay from surrounding farms, and fibre from the effluent clarifer (primary and secondary sludge). There is not that much information regarding what are the exact sources of the ashes but they are from various Alberta based sources. Particle size analysis and oxide composition are done on these ashes. The forestbased ashes were obtained after being burned at around 400-470°C.

3.3.2 Concrete mix design

Design stipulations are as follows: Compressive strength of 30 MPa at 28 days, maximum aggregate size of 8 mm and workability degree of 25-50 mm. The water-to-cement ratio was selected at 0.5. The mix design chosen for this study is shown in Table 3-1. Also superplastisizer was added to mix design to get the target slump between 25-50mm based on its standard instructions. The superplasticizer was a Glenium 3030 NS admixture, which meets ASTM C494.

Table 3-1- Mix Design

	Cement(kg/m ³)	Water(kg/m ³)	Sand(kg/m ³)	Coarse
				Aggregate(kg/m ³)
Mass/volume	400	200	600	1200

3.3.3 Preparation of specimens

Concrete cylinders 150 x 75 mm in diameter were cast. The coarse aggregate was first poured in the mixer, and then sand, cement and ash were added in that order

and mixed first as dry powder in the mixer for about two minutes. Subsequently, the design amount of water was added to the dry mixture and mixed in for a further two minutes. The assembled mould was filled with the cement concrete mix in two layers and compacted using a table vibrator. The cylinders were removed from the moulds after 24 hours of casting. They were then cured for 28 days. Figure 3-1 shows moulds, on the vibrator, being filled with concrete mix.



Figure 3-1- Moulds While Being Filled With Concrete Mix on Vibrator

For each mix prepared, slump and air content were evaluated as per ASTM C143 and ASTM C231 (pressure method) respectively. Figure 3-2 shows the slump and air content tests. The concrete slump test is an empirical test that measures the workability of fresh concrete. It refers to the ease with which the concrete flows. The air content test method covers the determination of the air content of freshly mixed concrete. The test determines the air content of freshly mixed concrete exclusive of any air that may exist inside voids within aggregate particles. Table 3-2, Table 3-3, Table 3-4 and Table 3-5 list these parameters for fresh concrete with the different amounts of each type of FBA. It can be seen that concrete mixes with FBA3 and FBA4 had decreasing slumps when the amount of ash was increased, which suggests that adding FBA 3 and FBA 4 decreases the workability of the concrete, whereas in case of mixes with FBA 1, the slump increased with the amount of ash introduced, which is promising for workability improvement. In the case of concrete containing FBA 2, there was no noticeable change at all in slump regardless of dosage, nor did the workability change that much. The air content as evaluated shows that adding FBA to concrete does not have a significant effect on the concrete. It is likely that forest-based ash does not interfere with the internal air-void network within concrete.



Figure 3-2- Slump Test and Air Content Test

	Slump(mm)	Air Content
Reference Mix	27	2.5
FBA 1-5%	24	2.7
FBA 1-10%	33	2.6
FBA 1-15%	32-33	2.6
FBA 1-20%	33	3.5

Table 3-2- Fresh Properties of FBA 1

Table 3-3- Fresh Properties of FBA 2

	Slump(mm)	Air Content
Reference Mix	27	2.5
FBA 2-5%	49	3.4
FBA 2-10%	35	4.5
FBA 2-15%	27	3.7
FBA 2-20%	25	3.7

	Slump(mm)	Air Content
Reference Mix	27	2.5
FBA 3-5%	40	3.4
FBA 3-10%	30	3.3
FBA 3-15%	27	3.8
FBA 3-20%	27	3.7

Table 3-4- Fresh Properties of FBA 3

Table 3-5- Fresh Properties of FBA 4

	Slump(mm)	Air Content
Reference Mix	27	2.5
FBA 4-5%	25	2.9
FBA 4-10%	20	3.3
FBA 4-15%	27	3.5
FBA 4-20%	20	3.5

The fresh properties of the different concrete mixes in Table 3-2, Table 3-3, Table 3-4 and Table 3-5 suggest that in concrete mixes with FBA 3 and FBA 4, there is a decrease in slumps when the percentage of FBA increases. A slump in the concrete mix with FBA 1 increases and with FBA 2 does not change that much. Tests of air content show that adding FBA to concrete does not change air content that much.

3.3.4 Compression and Tensile Tests

The compression and tension test setup employed in this study is shown in Figure 3-3. The concrete cylinder in the compression test is confined with a strain gauge with lateral and vertical LVDTs, which are connected to data acquisition equipment for recording the data through time. The compression force is applied by a MTS machine until the concrete cylinder develops obvious cracks. The splitting tensile test is done by a Forney machine and the force is applied, gradually, on the side of the concrete cylinder until the cylinder cracks and breaks. Forney machine shows the breaking load which will result in the tensile strength through related calculations based on ASTM C496.



Figure 3-3- Compression and Tensile Tests

3.4 **Results and discussions**

3.4.1 Compressive strength

Figure 3-4 shows the photo reproduction of the equipment used for compression testing. The tests were conducted in accordance with ASTM C496 (2011). The results are in Table 3-6,

Table 3-7, Table 3-8, Table 3-9 and Table 3-10. This data is further depicted graphically in Figure 3-5 below.



Figure 3-4- Compression Test

Table 3-6- Modulus of Elasticity and Poisson's Ratio of Concrete Mixes with Different Ashes

FBA Percentage	E(MPa),v	E(MPa),v	E(MPa),v	E(MPa),v
	(FBA1)	(FBA2)	(FBA3)	(FBA4)
0	33543, 0.21	33543, 0.21	33543, 0.21	33543, 0.21
5	20327, 0.20	19727, 0.20	22238, 0.20	23540, 0.20
10	16160, 0.14	20324, 0.18	17938, 0.19	20183, 0.17
15	18335, 0.17	16814, 0.16	19248, 0.21	19300, 0.22
20	18387, 0.19	13617, 0.16	18432, 0.19	18532, 0.20

Table 3-7- Compressive Strength of FBA 1 Concrete Cylinders after 1, 3,7,14 and 28 Days

Percentage of FBA	Average Compressive	Average Compressive Strength	Average Compressive Strength	Average Compressive Strongth	Average Compressive Strength
(70)	(N/mm^2) 1 st day	(N/mm^2) $3^{rd} day$	(N/mm^2) $7^{th} day$	(N/mm^2) 14 th day	(N/mm^2) $28^{th} day$
0	17.36	27.27	32.24	33	33.14
5	11.75	20.36	25	31.74	27.92
10	5.69	15.76	18	22	24.5
15	13	17	23.82	26.74	28.57
20	9	10	17.78	19.25	22.75

Percentage of FBA (%)	Average Compressive strength (N/mm ²) 1 st day	Average Compressive Strength (N/mm ²) 3 rd day	Average Compressive Strength (N/mm ²) 7 th day	Average Compressive Strength (N/mm ²) 14 th day	Average Compressive Strength (N/mm ²) 28 th day
0	17.36	27.27	32.24	33	33.14
5	9.5281	16	21.78	22.48	24.55
10	6.32	19.88	24.26	23.65	27.12
15	15.31	16.6	20.5	22.55	23.5
20	10.3	11.82	14.46	14.31	16.3

Table 3-8- Compressive Strength of FBA 2 Concrete Cylinders after 1, 3,7,14 and 28 Days

 Table 3-9- Compressive Strength of FBA 3 Concrete Cylinders after 1, 3,7,14 and 28 Days

Percentage	Average	Average	Average	Average	Average
of FBA	Compressive	Compressive	Compressive	Compressive	Compressive
(%)	Strength	Strength	Strength	Strength	Strength
	(N/mm^2)	(N/mm^2)	(N/mm^2)	(N/mm^2)	(N/mm^2)
	1 st day	3 rd day	7 th day	14 th day	28 th day
0	17.36	27.27	32.24	33	33.14
5	12.18	21.79	27.91	28.04	35
10	11.5	23.57	25.35	29.6	31.72
15	12.5	20.54	24	25.84	30.33
20	10.27	18.1	20	22.51	25

Table 3-10- Compressive Strength of FBA 4 concrete cylinders after 1, 3,7,14 and 28 days

Percentage	Average	Average	Average	Average	Average
of FBA	Compressive	Compressive	Compressive	Compressive	Compressive
(%)	Strength	Strength	Strength	Strength	Strength
	(N/mm^2)	(N/mm^2)	(N/mm^2)	(N/mm^2)	(N/mm^2)
	1 st day	3 rd day	7 th day	14 th day	28 th day
0	17.36	27.27	32.24	33	33.14
5	14	28	31	34	42.5
10	16.52	24.12	28.18	32.5	38.3
15	14.81	24.7	27	29.83	29
20	12.58	22	22.28	27.75	30



Figure 3-5 Variation of Compressive Strength with Different Ashes at 1, 3, 7, 14 and 28 Days





Figure 3-6 shows the variation in 28-day compressive strength with varying dosages of FBA. The optimum percent of ash in each type of FBA could be identified. From the above figure, one can easily see that FBA 4 provides the

greatest strength at lower dosages of 5 to 10%. In fact even at 15% cement replacement, the strength is same as that of the reference concrete. Hence, one can conclude that this particular FBA can be used effectively up to 15% to get other benefits of a pozzolanic admixture without sacrificing the strength. Notice that concrete made with FBA 3 has almost the same compressive strength as that of the reference concrete when up to 15% of the cement is replaced. Again, it appears that replacing 15% of the cement is the optimum dosage for FBA 3.

However in the case of FBA 2, the strength drops considerably at 5% dosage and picks up at 10% dosage. In the case of FBA 1, there was a continuous drop up to 10% dosage but the compressive strength picked up thereafter. At the 15% dosage, the strength was the same as that of the reference mix. To summarize, the four FBAs sampled in this study appear to reach an optimal dosage at between 10-15% of cement replacement.

Considering the other parameters here, namely the modulus of elasticity and poisson's ratio, together with the strength, the sample of FBA 4 shows the best results. From these ashes' physical and chemical characterization, this superior performance in sample FBA 4 may be attributed to its higher CaO content (which will bring hydraulic effect to concrete) and finer particle size distribution compared to the three other types of FBA examined in this study. At the other end of the performance spectrum lies FBA 2, which generally showed the least strength for corresponding dosages compared to the other three ash samples. Once again, this may be attributed to the CaO content (lowest among the four ash samples) and particle size distribution (the coarsest of all).

3.4.2 Tensile strength

The split tensile strength of the specimens was ascertained as per ASTM C496 (2011). Table 3-11,

Table 3-12, Table 3-13 and

Table 3-14 show the test results for the four types of FBA (1 through 4), respectively. The tables show the tensile strength at 1, 3, 7, 14 and 28 days of curing. Figure 3-7 shows the tensile test underway using the Forney machine. Further, Figure 3-8 is a plot of these results to highlight the variation with age at each dosage level, while in Figure 3-9, the results are illustrated specifically for 28-day strength.



Figure 3-7 Tensile Test

Percentag e of FBA (%)	Average Tensile strength (N/mm ²) 1 st day	Average Tensile Strength (N/mm ²) 3 rd day	Average Tensile Strength (N/mm ²) 7 th day	Average Tensile Strength (N/mm ²) 14 th day	Average Tensile Strength (N/mm ²) 28 th day
0	2.04	3.26	3.52	3.57	3.63
5	1.75	2.71	3.24	3.36	3.56
10	1	2.31	2.61	2.75	2.83
15	0.8	2.5	2.58	2.59	3.1
20	0.7	1.89	2.17	2.16	2.49

Table 3-11- Tensile Strength of FBA 1 Concrete Cylinders after 1, 3, 7, 14 and 28 Days

Table 3-12- Tensile Strength of FBA 2 Concrete Cylinders after 1, 3, 7, 14 and 28 Days

Percentage of FBA (%)	Average Tensile strength (N/mm ²) 1 st day	Average Tensile Strength (N/mm ²) 3 rd day	Average Tensile Strength (N/mm ²) 7 th day	Average Tensile Strength (N/mm ²) 14 th day	Average Tensile Strength (N/mm ²) 28 th day
0	2.04	3.26	3.52	3.82	3.63
5	1.38	2.48	2.58	2.93	2.97
10	1.59	2.98	2.43	2.97	2.96
15	1.84	1.87	2.62	2.68	2.28
20	1.41	1.65	2.33	2.19	1.8

Percentage of FBA (%)	Average Tensile strength (N/mm ²) 1 st day	Average Tensile Strength (N/mm ²) 3 rd day	Average Tensile Strength (N/mm ²) 7 th day	Average Tensile Strength (N/mm ²) 14 th day	Average Tensile Strength (N/mm ²) 28 th day
0	2.04	3.26	3.52	3.82	3.63
5	1.88	2.64	2.97	2.81	3.16
10	1.87	2.93	3	3.2	3.04
15	1.62	2.31	2.46	2.3	3.08
20	1.44	2.1	2.18	2.12	2.89

Table 3-13- Tensile Strength of FBA 3 Concrete Cylinders after 1, 3, 7, 14 and 28 Days

Table 3-14- Tensile Strength of FBA 4 Concrete Cylinders after 1, 3, 7, 14 and 28 Days

Percentage of FBA (%)	Average Tensile strength (N/mm ²) 1 st day	Average Tensile Strength (N/mm ²) 3 rd day	Average Tensile Strength (N/mm ²) 7 th day	Average Tensile Strength (N/mm ²) 14 th day	Average Tensile Strength (N/mm ²) 28 th day
0	2.04	3.26	3.52	3.82	3.63
5	1.61	2.81	2.67	3.22	3.61
10	1.67	2.64	2.89	3.15	3.63
15	1.4	2.43	2.54	2.91	3.34
20	1.53	2.34	2.74	2.96	3.49



Figure 3-8- Variation of Tensile Strength with Different Ashes after 1, 3, 7, 14 and 28 Days





From the figures above, it can be easily seen that the tensile strength of concrete with FBA 4 remains more or less constant over a wide range of dosages (i.e., up to 20% cement replacement), whereas in the case of the other three types of FBA, the strength drops to various levels with an increase in the amount of FBA.

Through comparison of concrete with these different forest based ashes, the drop in strength for concrete containing FBA 2 was the greatest, as reflected in the entire dosing range from 5 to 20%.

Sample FBA 4 contains predominantly CaO as discussed in the previous chapter, which makes the concrete stronger even when high dosages are used due to its hydraulic effect in concrete. Another reason that concrete with FBA 4 has better compressive and tensile strength is that the ash has very fine particles. For the same reason, FBA 2 performs poorly because it has a low CaO content and a very coarse particle-size distribution - the coarsest of all of the FBAs.

There is not that much certainty regarding what are the exact sources of the ashes but they are from various Alberta based sources. Particle size analysis and oxide composition are done on these ashes to know more about them. Adding FBA 3 and FBA 4 decreases the workability of the concrete, whereas adding FBA 1 increased the workability. In the case of concrete containing FBA 2, there was no noticeable change at all in the slump and, consequently, in the workability. The results of an air content test on fresh concrete with FBA showed that it is likely that FBA does not interfere with concrete's internal air-void network.

Based on ASTM requirements, results show that the sum of silica, alumina and iron oxides in FBA 1, 2, 3 and FBA 4 is lower than the minimum allowable percent and ashes' value of Alkalis is more than its allowable maximum limit. In other words, the ashes don't meet the minimum requirements of ASTM, but regarding the findings of Zerbino et al. (2012), if the ash particles become smaller than 75 microns, the potential of alkali silica reaction would be less.

3.5 Conclusion:

FBA was added to the concrete mix as a supplementary material instead of cement. First, the ashes were chemically and physically studied and then fresh and hardened properties of FBA concrete were studied. Based on the observations made in this study, the specific findings were made:

- From the physical and chemical characterization of previous chapter, FBA
 4 delivered the best performance, because of its higher CaO content and
 finer particle size distribution compared to the other three types of FBA
 examined in this study. This suggests that FBA 4 will deliver a better
 strength performance both by acting as filler and as pozzolanic material.
- 2. Considering the modulus of elasticity and poisson's ratio together with the tensile and compressive strength, FBA 4 was at this stage in the study thought to become the best suited of the four FBA samples for use as pozzolanic replacements to cement and FBA 2 generally showed the least strength characteristics.
- The four FBAs sampled in this study appear to reach an optimal dosage at between 10-15% of cement replacement.
- 4. Regarding ASTM requirements, results from previous chapter show that sum of silica, alumina and iron oxides FBA1, 2, 3 and FBA 4 is lower than minimum allowable percent and the ashes value of Alkalis is more than their allowable maximum limit. So the ashes don't obey minimum requirements of ASTM and there would be the risk of alkali silica reaction but regarding findings of other researchers, if the ashes particles become

smaller than 75 microns, the potential of alkaline silica reaction would be less.

5. In order to overcome risk of alkali silica reaction having smaller particle size of ashes is recommended and ash with more CaO content and smaller particle size would be good criteria for FBA as supplementary materials in concrete to bring sufficient properties.

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Chapter 4. SCBA as a Pozzolanic Admixture in Concrete for Resistance to Sustained Elevated Temperatures³

4.1 Abstract

In the present investigation, a feasibility study is made to utilize the sugar cane bagasse ash (SCBA) as an admixture in concrete and examine its role in imparting resistance under elevated temperatures. The ash was obtained from a sugar mill in India where the bagasse was recycled as fuel for the mill. This ash was characterized for its physical properties and chemical composition. Incorporated as a supplementary cementing admixture, SCBA replaced Portland cement from 0 to 25% by mass fraction at 5% increments. The resulting concrete was subjected to elevated temperatures of 300°C, 400°C and 500°C, exposed for 2 hours in each case. A reference series was examined at room temperature. Compressive and flexural strength were evaluated and compared with the reference performance at room temperature and reported as residual properties. The results show that the SCBA sample had a grain size distribution very similar to that of the Type GU Portland cement used in this study. X-ray fluorescence showed that this ash was chiefly composed of SiO2 (>70%). The compressive strength of concrete cubes increases up to 10% SCBA incorporation. Even at 15% cement substitution, strength matches that of the reference mix containing Portland cement alone. While there was a consistent drop in the compressive strength at higher temperatures, including SCBA marginally slows down this deterioration. The flexural strength of concrete containing SCBA was always lower than that seen

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with Portland cement alone. Once again, the drop was seen to be less significant up to 10% cement substitutions. The findings strongly endorse that bagasse ash imparts resistance to concrete against elevated temperatures and may be used as a supplementary cementing admixture.

4.2 Introduction

Bagasse is currently used as a biofuel in the pulp and building materials industry. It is found that for every 10 tons of sugar cane crushed in a sugar mill, generally about 3 tons of bagasse is produced. In most cases, this bagasse is recycled as fuel within the sugar mill. Once it is ground to suitable fineness, there is immense potential to use this bagasse ash as a partial replacement for cement in cement-related works since it is cost effective. Since bagasse is a by-product of the sugar cane industry, the quantity of production in each country is in line with the quantity of sugarcane produced. Sugar cane/sugarcane can be ground to requirements and burnt and used as a cement replacement in concrete. Figure 4-1 shows the sugarcane bagasse sourced for this study.



Figure 4-1 Source: Pandavapura Sugar Factory

There has been considerable interest lately in agro-sourced ash for cement replacement. Among the agro-sourced ashes, rice-husk ash and various types of wood ash have been examined by Ganesan et al. (2008), Rao et al. (2012), and Paula et al. (2010). In the past decade, researchers have undertaken studies on SCBA, focusing on its feasibility as a supplementary cementitious material and its influence on the hardened properties of Portland cement concrete.

Paula et al. (2010) examined cement mortars with up to 20% substitution of Portland cement with SCBA. They have reported that there is a general improvement in the strength of concrete and therefore SCBA is a viable substitute. Fairbairn et al. (2012) examined the chemical, mechanical and thermal properties of concrete with different levels of substitution of cement by SCBA. The results have been reported to be favorable. The results show that using SCBA reduces CO_2 emissions by using less cement content and improves the strength of the concrete. Casuldelli et al. (2013) examined SCBA-mixed cement mortars and pastes and found favorable results with regard to alkali-activated pastes and mortars. Chusilp et al. (2009) found a reduction in the heat of hydration as well as a reduction in water permeability without much change in strength. Srinivasan et al. (2010) conducted experiments replacing Portland cement with 5 to 25 % of SCBA and found similar results. Radke et al. (2012) found a slight increase in strength when substituting up to 20% SCBA in cement. Amin et al. (2010) also found that the optimal replacement ratio for bagasse ash in pozzolan is 20%. This percentage reduces chloride diffusion by more than 50% and has no adverse effects on other properties of the hardened concrete. Bagasse ash is a valuable

pozzolanic material that has the potential to reduce costs, conserve energy, and minimize waste emission (Wild et al. 1996). Paula et al. (2010) conducted tests on SCBA and the results prove that SCBA can be effectively replaced up to 10% wherein the strength does not decrease that much. Kawade et al. (2013) reported a small increase in strength with SCBA up to 15% cement substitution. Similar results were reported by Hailu et al. (2012). The increase in strength may be attributed to the transformation of the CH phase into the CSH phases upon the pozzolanic reaction, as reported by Muangtong et al. (2013). In addition to the increase in strength, there is some improvement to durability as noted by Ruckzon et al. (2012), who found that the SCBA addition increases the resistance to chloride penetration. Some concerns regarding fresh properties have been noted. These concerns, noted by Hailu et al. (2012) include a higher normal consistency and a longer setting time. When bagasse ash is increased, the workability of the concrete is diminished slightly.

While the available literature summarized above recognizes SCBA's potential as a supplementary cementitious material, these studies have been confined to the effects of SCBA on concrete at room temperature. Note that in the case of concrete products like normal concrete, high-strength concrete and concrete with other pozzolans like rice husk ash and fly ash, mechanical behavior was studied under elevated temperatures. Arioz et al. (2007) studied the effects of elevated temperatures on properties of concrete and noted that the surface cracks became visible when the temperature reached 600°C. The cracks were very pronounced at 800°C and increased extremely when the temperature increased to 1000°C. Concrete specimens subjected to temperatures of 1200°C were completely decomposed. Srinivasa et al. (2004) studied the variation of compressive strength of high-strength concrete at elevated temperatures and found that an increase in compressive strength gradually takes place between the temperature ranges of 100°C and 350°C. The concrete retains its original strength up to a temperature of 400°C for all durations of exposure. The rate of decrease in compressive strength is gradual in the range of 400°C to 700°C. Pathan et al. (2012) concluded that when the temperature is increased to 300°C, the compressive, split tensile, flexural strength and impact strength of specimens is decreased for conventional and high-strength concrete. Husem (2006) studied the effects of high temperatures on the compressive and flexural strength of ordinary and high-performance concrete and concluded that the flexural and compressive strength of these types of concrete decreases when the temperature increases. Rao et al (2011) studied the replacement of RHA in the range of 5% to 20% and found that replacement does not change the compressive strength of concrete. The residual compressive strength of concrete for all the RHA replacements increases at the initial temperatures of 100 – 150 °C and thereafter decreases gradually up to 700 °C because pore water evaporates at 1000°C and the concrete matrix becomes brittle. A 15% replacement of RHA is found to be optimal, because the residual compressive strength at various temperatures in the range of 100 - 700 °C shows similar strength to that of concrete without RHA at 28 days. An overall performance of 10% RHA replacement level at both 7 and 28 days shows better residual compressive strength than that of normal concrete. Harison et al. (2014) found that a loss in compressive strength occurs with a rise in temperature, and that the measured compressive strengths were normalized with respect to room temperature values. Results indicated the losses in relative strength were due to high-temperature exposure and the presence of 10% silica fumes as a cement replacement seemed to have no significant effect.

As noted above, studies have largely focused on the behavior of normal concrete, high-strength concrete, and concrete with rice husk ash and fly ash at high temperatures. Based on previous investigations, none of the studies examined for this thesis looked at how SCBA affects the performance of concrete at high rather than normal temperatures

This project aims at examining how suitable and effective SCBA is when it is used as a supplementary cementing admixture under an elevated temperature. The purpose of this project is to study variations of the compressive strength of cement mortar and cement concrete cubes and flexural strength of cement concrete beams subjected to sustained elevated temperatures. In this study, SCBA is used as a partial cement replacement in concrete specimens, with percentages varying from 0 to 25 (i.e., 0%, 5%, 10%, 15%, 20%, 25%) under elevated temperatures of 300°C, 400°C and 500°C for 2 hour exposure is done.The performance of the concrete, specifically its residual compressive and flexural strength, is then studied.

4.3 Experimental Program

As noted above, six mixes were designed for this study. For the compression and flexural tests, four sets of cubes were cast for every mix. One set was left to
remain at room temperature, while the other three sets were subjected to temperatures of 300°C, 400°C and 500 °C for duration of two hours each.

4.3.1 Materials

Ordinary Portland cement conforming to IS 269: 1976, locally available dry sand and crushed granite aggregate conforming to IS 383-1970 were used in this study. The grading of sand conforms to IS 383-1970 zone II grading while the coarse aggregate had a maximum particle size of 20 mm. The SCBA was sourced from a sugar factory in Pandavapura (near Mysore, India). It was ground to fineness similar to that for the Portland cement examined in this study.

4.3.2 Physical and Chemical Examination of SCBA

The particle-size distribution for the SCBA was done using a particle-size analyzer named Mastersizer by the Malvern Company. In order to characterize the mineral composition in the ash, the ash was subjected to Energy Dispersive X-ray (EDX), X-ray diffraction (XRD) and X-Ray Fluorescence (XRF) testing. To illustrate the ash's morphology, samples were examined under a scanning electron microscope (SEM).

4.3.3 Concrete mix design

The concrete mixes were designed based on achieving a 28-day compressive strength of 20 MPa. The desired workability was met with a slump between 25-60 mm. The mix proportions are shown in Table 4-1. The specimens were demolded after 24 hours and cured in a water bath for 28 days. The SCBA ash was added as

a supplementary material instead of cement while mixing the materials in the mixer.

Table 4-1- Mix Design

	Fine Aggregate	Coarse Aggregate	Cement	Water
Kg/m ³	739	1110	367	183

4.3.4 Preparations of specimens

4.3.4.1 Preparation of cement concrete specimens

Cement concrete cubes of 150 x 150 x 150 mm and cement concrete beams of 500 x 100 x 100 mm were cast. The cubes and beams were cast in accordance with IS 516:1959. Oil was applied along the inner surface of the metal molds. The coarse aggregate was spread evenly on a large tray. Sand and cement was dry-mixed in the mixer and spread evenly over the coarse aggregates. The dry ingredients were then hand-mixed thoroughly for about 3 min. Estimated amount of water was added to the dry mixture and mixed for 4 min. The assembled mold was filled with the cement concrete mix in three layers and compacted using a table vibrator and a needle vibrator. The cubes were removed from the molds after 24 hours of casting and cured for 28 days. Figure 4-2 shows the molds after being filled with a cement mortar mix and mechanically compacted.



Figure 4-2 Moulds after Being Filled with Cement Mortar Mix and Mechanically Compacted

4.3.4.2 Exposing specimens to elevated temperatures

A furnace with the capacity of reaching a maximum temperature of 1000 °C was used to conduct the experiments. The specimens were subjected to elevated temperatures of 300° C, 400° C and 500° C with an exposure time of two hours for each temperature. These specimens were placed in an electric furnace 2.1 x 1.1 x 1m. This furnace is heated with an electric coil and is capable of attaining a maximum temperature of 1000° C. The samples were exposed to the required temperature and for the required duration. The heat was turned off, the top sliding cover of the electric oven was opened, and the samples were allowed to cool in the oven. After the surface was cooled for ten minutes, the temperature was measured with the help of a non-contact thermometer and recorded. When the oven cooled down to the ambient temperature, the specimens were taken out and stocked for future testing. Figure 4-3 shows the furnace used.



Figure 4-3 Furnace with Capacity of 1000°C

4.3.4.3 Compression and flexural tests

Figure 4-4 shows the compression and flexural instruments that were used. The flexural test is a 3-point load test and it applies load on the beam gradually to reach failure in the beam starting from cracking to crushing. The compression instrument also applies load on the cube gradually from the top surface to reach crack and crush.



Figure 4-4 Compression and Flexural Tests

4.4 Results and analysis

4.4.1 Physical Properties and Chemical Composition of SCBA

Table 4-2 and Table 4-3 show the elemental and oxide composition for SCBA according to results from the EDX analysis and XRF testing. Figure 4-5 shows the particle-size distribution of SCBA and it confirms that the grains were similar in size and distribution to what was found in Portland cement, as intended. In SCBA, the distinct peaks relate to quartz, cristobalite and microcline.

Elements	Weight
	Percentage
0	46.83229
Na	0.374375
Mg	3.662789
Al	3.13841
Si	22.40981
Р	4.082293
Cl	0.012102
K	8.842095
Ca	2.051244
Ti	0.279344
Fe	2.707545

Table 4-2 EDX Test Results of SCBA

Table 4-3 (Oxide C	omposition	of SCBA
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Oxide	Average (wt %)			
Sio ₂	71.62			
MgO	6.84			
P ₂ O ₅	6.27			
K ₂ 0	5.97			
Al ₂ o ₃	3.42			
CaO	2.32			
Na ₂ o	1.53			
Fe ₂ o ₃	1.28			
So ₃	0.43			
Tio ₂	0.09			
Cr ₂ o ₃	0.08			
MnO	0.06			
SrO	0.03			
CuO	0.02			
Zro ₂	0.02			
ZnO	0.01			
Rb ₂ o	0.01			
Ni ₂ o ₃	0.01			



Figure 4-5 Particle-Size Analysis of SCBA and OPC



Figure 4-6 XRD Test of SCBA

Chemical analysis on SCBA showed that silica makes up more than 70% of the oxide components in the ash. The total value of alkalis (K_2O+Na_2O) was more than 6%, which immediately raises a concern about the potential for an alkaline-silica reaction. However, Zerbino et al. (2012) reported that although alkali content may exceed the allowable limits stipulated in ASTM C618 (2012), the potential for alkali-silica reactivity is strongly dependent upon the particle size of the ash. In their study on RHA, Zerbino et al. (2012) reported that so long as the entire ash passed through the no. 200 sieve (75 microns), there was no alkali-silica reactivity even with more than 5% alkali (K_2O+Na_2O). As noted in Figure 4-5, the SCBA used in this study was in its entirety finer than 100 microns. Therefore, based on the above, the expectation is that although the alkali content was 7.5%, for the relatively small particle size precludes any potential alkali-silica reaction.

It is clear from the chemical and physical characteristics of sugar cane bagasse ash, as illustrated in previous figures and tables, that the dominant oxide in that is silica. Results from the EDX and XRF analyses further confirm that silica is the predominant acidic oxide (>70%). It is worth noting that amorphous silica, apart from being effective as a pozzolanic material, also possesses favorable thermal properties, notably an extremely low coefficient of thermal expansion against crystalline silica.

ASTM C618 (2012) requires a minimum of 70% for the sum of silica, alumina and iron oxides. It also specifies a maximum limit of 5% for SO₃, 12% for unburnt carbon and 1.5% for alkalis such as Na₂O and K₂O. The identification of oxides in SCBA also reveals that this ash totally contains over 6% for K₂O and Na₂O, and exceeds the limit prescribed by ASTM C618 for pozzolanic admixtures. Clearly, this raises concerns about the potential alkali-silica reactivity and resultant loss in durability in the long-term. The size of the particles in the sampled SCBA was between 0 and 100 micrometers, as seen in Figure 4-5. Figure 4-7 illustrates the shape of different particles in the ash, as seen with the SEM. The shapes may be described as spongy (Figure 4-7a), spherical (Figure 4-7b) and book-like (Figure 4-7c). Traces of unground charcoal were also seen (Figure 4-7d), suggesting that further grinding was necessary.



Figure 4-7- SEM Test of SCBA

4.4.2 Compressive strength

The compressive strength was determined from concrete cubes at 28 days. As stated earlier, the values in the case of those specimens subjected to elevated temperature represents a residual strength.

Sl.no	Percentage Replacement of SCBA (%)	Average Compressive Strength (N/mm ²) under room temperature	Average Compressive Strength (N/mm ²) under 300°C	Average Compressive Strength (N/mm ²) under 400°C	Average Compressive Strength (N/mm ²) under 500°C
1	0	43.55	40	35.2	29.11
2	5	45.6	43.3	37.6	29
3	10	46.4	43.5	38.53	30.1
4	15	44.62	41.64	37.2	28.57
5	20	38.53	36.88	32.44	25.77
6	25	29.7	28.59	25.19	20.14

Table 4-4 Compressive Strength of SCBA Concrete Cubes after 28 Days under Different Temperatures



Figure 4-8- Variation of Compressive Strength under Different Temperatures for a two-hour Duration

Figure 4-8 shows that concrete made with SCBA retains its strength up to a cement replacement of 15%. For comparison, studies using rice husk ash to gauge concrete resistance to elevated temperatures show an optimum performance at 15% cement replacement (Rao, R. 2012). It was found that strength increases initially up to a cement replacement of 10% and strength decreases through increasing percentage from 10 to 15 and gets the initial strength with zero percentage. It was found that there would be almost the same initial strength up to 30% replacement for fly ash and drops up to reaching the initial strength at cement replacement of 15% (Shang, S.H. 2013).



Figure 4-9- Variation in Compressive Strength

It is inferred from Figure 4-9 that with an increase in the sustained temperature, there is a gradual decrease in compressive strength of the specimens, irrespective of what percentage of SCBA is being used to replace cement. However, in all mixes, there was only a slight drop in residual strength up to 300 °C. This has been previously reported for rice husk ash as well (Rao and Tummalapudi, 2012). Also, when the residual strength of a mix is normalized with respect to its compressive strength at room temperature, it is seen that the rate of drop in strength is lower with an increase in the ash-to-binder ratio.

4.4.3 Flexural strength results

The flexural strength test on the specimen was carried out as per IS 516:1959 using the flexural strength testing machine adopting four-point loading. The residual flexural strength is reported in Table 4-4.

Sl.no	Percentage Replacement of SCBA (%)	Average Flexural Strength (N/mm ²) under Room Temperature	Average Flexural Strength (N/mm ²) under 300°C	Average Flexural Strength (N/mm ²) under 400°C	Average Flexural Strength (N/mm ²) under 500°C
1	0	1.77	1.60	1.35	1.08
2	5	1.62	1.43	1.21	0.95
3	10	1.50	1.33	1.12	0.90
4	15	1.36	1.20	0.99	0.82
5	20	1.20	1.12	0.9	0.73
6	25	1.06	0.94	0.76	0.63

Table 4-4 Flexural Strength Results under Different Temperatures

Figure 4-10 shows that when the ash content is increased, the flexural strength of concrete made with SCBA will drop. Unlike its compressive strength, which showed an improvement in its residual strength up to 10% and sometimes even 15% cement replacements, the flexural strength showed a consistent drop at all temperatures of exposure. Nevertheless, as with the compressive strength, there was a sharp drop in residual flexural strength beyond 300°C, as shown in Figure 4-10b.







Figure 4-11 Flexural Strength Variation

Figure 4-11 shows that there is a gradual decrease in flexural strength with an increase in the percentage replacement of SCBA in concrete. Also, across the mixes, when the specimens were subjected to sustained elevated temperatures of 300°C, 400°C and 500°C, there was a significant decrease in the flexural strength and this drop ranged from 20% to 40% with respect to room temperature. This drop is very similar to that seen with concrete containing fly ash and subjected to

high temperature in an identical range (Shang, 2013). From the graph above, it can be inferred that the drop in strength in plain cement concrete cubes is more when compared with the concrete with bagasse ash. Nevertheless, as with the compressive strength, there was a sharp drop in residual flexural strength beyond 300°C, as shown in Figure 4-11b.

4.5 Conclusion

- Chemical tests on SCBA show that silica is the most predominant oxide (more than 70%). The particle-size distribution is nearly the same as that of Portland cement, ranging from 1 to 100 microns. A range of particle shapes was identified, including spongy, spherical and book-structure.
- The effect of SCBA on the strength of concrete was examined with a particular focus on the resistance to sustained elevated temperatures up to 500 °C. Based on the compression and flexural tests, SCBA appears to impart benefits similar to those of other industrial and agro-sourced ash in high temperature applications.
- When examined at room temperature, the compressive strength of concrete dosed with SCBA increases up to a cement substitution of 10%. It was seen that with an increase in the sustained temperature, there was a gradual decrease in compressive strength of the specimens, irrespective of the cement percentage which was replaced by SCBA. However, in all mixes, there was only a slight drop in residual strength, up to 300 degrees. Also, when the residual strength of a mix was normalized with respect to its compressive

strength at room temperature, it was seen that the rate of drop in strength was lower with an increase in the ash-to-binder ratio.

• At room temperature, there was a gradual decrease in flexural strength with an increase in the percentage replacement of SCBA in concrete. Also, when the specimens were subjected to sustained elevated temperatures, there was a significant decrease in the strength and this drop ranged from 20% to 40%. It was inferred from results that the drop in strength in concrete containing cement alone was more than that concrete containing bagasse ash.

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Chapter 5. Conclusions and recommendations

5.1 Conclusions

- It was shown in x-ray diffraction charts that in ashes (FBA 1 through 4), the chemicals portlandite, arcanite, quartz and calcite exhibited distinct peaks compared to the other minerals. Also, in sugar cane bagasse ash, it was noticed that the distinct peaks belong to quartz, cristobalite and microcline.
- Tests of scanning electron microscope, energy-dispersive x-ray spectroscopy and particle-size analysis were done on ashes. Sizes of the particles in FBA 1, FBA 2, FBA 3 and FBA 4 were more than 100 micron while the particle-size distribution of SCBA is nearly the same as that of Portland cement, ranging between 1 to 100 microns. FBA 4 has more of the finer particles, while FBA 2 has the coarsest particle-size distribution. A range of particle shapes were identified for FBAs and SCBA including spherical, book-structure, pin-like, and spongy.
- With respect to different FBA mineral compositions, it is inferred that in FBA 4, the CaO content is more than the other ashes, which may potentially impart a latent hydraulic effect. Chemical tests on SCBA show that silica is the most predominant oxide (more than 70%).

- Considering the modulus of elasticity and Poisson's ratio together with the tensile and compressive strength, FBA 4 was the best suited of the four FBA samples for use as a pozzolana due to its higher CaO and finer particle size, which results in the pozzolanic and filler effect. FBA 2 was the least suited. The four forest-based ash types sampled in this study appear to reach an optimal dosage of ash at between 10 and 15% of cement replacement.
- The effect of SCBA on the compressive and flexural strength of concrete was examined under sustained elevated temperatures up to 500 °C. Increasing the sustained temperature will result in a gradual decrease in the compressive strength of the specimens. However, it was seen that there was only a slight drop in residual strength up to 300 °C. While specimens were subjected to sustained elevated temperatures, there was a significant decrease in flexural strength. This drop ranged from 20% to 40%. This suggests that SCBA imparts benefits similar to those imparted by other industrial and agro-sourced ash at high temperatures.
- Considering both SCBA and FBAs for the risk of silica alkali reaction and regarding ASTM requirements, FBAs and SCBA don't obey minimum requirements of ASTM and there would be the risk of alkali silica reaction but regarding findings of other researchers, if the ashes particles become smaller than 75 microns, the potential of alkali silica reaction would be less. SCBA particles are smaller than 75 microns, so the risk for alkali

silica reaction would be lower. To avoid risking an alkali silica reaction, it is advised to use smaller ashes and ashes with more CaO.

• Needless to say that, comparing the effect of the forest based ash and sugar cane bagasse ash in concrete, assuming in the same conditions, sugar cane bagasse ash will bring more acceptable strength while bringing less concerns of sulphate attack and alkali silica reaction. This distinction may be attributed to the following reasons: Sugarcane bagasse ash contains more silica which will result in stronger concrete while forest based contains more CaO which although rendering it latently hydraulic in concrete will likely not result in similar strength like a silica-rich pozzolan. Also, sugarcane bagasse ash contains less Na₂O, K₂O and SO₃ which brings a lower probability of alkali silica reaction and sulphate attack in comparison with forest based ash. Thirdly, sugar cane bagasse ash contains much finer particles in comparison with forest based ashes which will result in higher strength in concrete.

5.2 Recommendations

- As it was noted, there may be concerns of sulphate attack and alkali silica reaction attack which will lead to expansion, cracks and sooner failure in concrete. In order to overcome these problems, tests of sulphate attack and ASR attack are recommended while usage of finer particle ashes is recommended.
- As the amorphous and crystal phase of the silica in ashes is not that clarified in the test, it's recommended for further tests.

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